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OXIDATION REDUCTION POTENTIAL OF THE CU(III)/CU(II)
SYSTEM IN ALKALINE SOLUTIONS

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OXIDATION-REDUCTION POTENTIAL OF THE CU(III)/CU(II)
SYSTEM IN ALKALINE SOLUTIONS

G. I. Rozovskiyy and A. K. Misyavichios

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Abstract

Potentials of barium and sodium cuprate coatings, obtained by an electrochemical method on platinum, and in isolated cases, on copper, were measured. It was shown that the stationary potentials were the same in the case of both cuprates.

In 17-0.1 N NaOH solutions, the following dependencies were established: at 5°C $E = 0.77 - 0.52 \lg a_{OH^-}$, at 25°C $E = 0.74 - 0.060 \lg a_{OH^-}$. In 10^{-1} - 10^{-3} N NaOH solutions, the slope coefficient for a linear dependence of the potential on pH is equal to $(3/2) \cdot (2.303RT/F)$. Upon addition of cuprite, the measured stationary potential complies with the dependence $E = E_1 - (2.303RT/F) \lg a(c_{Cu(II)}^* + c_{Cu(II)}) - (2.303RT/F) \lg a_{OH^-}$, where $c_{Cu(II)}$ is the concentration of the added divalent copper, and $c_{Cu(II)}^*$ is the concentration of the species formed upon reduction of trivalent copper.

Possible variations of potential determining reactions were discussed.

Article

The values of oxidation potentials of trivalent copper, computed on the basis of indirect data [1-4], as well as those obtained by means of an analysis of potential-time curves [5, 6], vary greatly.

Two works are known in which the results of direct measurements of potentials of some compounds of trivalent copper are presented. Columbo [7] measured the potentials of tellurate and periodate solutions of trivalent copper. Dailey [8] measured potentials of trivalent copper compounds in 0.005-1 N NaOH, based on the opinion of [9] on potentials of

barium cuprate.¹ Measurements of potentials in work [8] occurred upon mechanical contact of a platinum electrode with a mixture containing barium cuprate (less than 15%), copper oxide, and also, apparently, barium and sodium hydroxide.

The electrochemical methods developed by us previously for the synthesis of cuprates [10, 11] allowed the contact of a solid cuprate with an indicator electrode necessary for measuring potentials by means of anodic deposition of cuprates on platinum and copper.

In this work, the effect of the alkalinity of the medium on the stationary potential of sodium and barium cuprates in the range of 10^{-3} - 10^{-7} N NaOH, additions of cuprite, and temperature was studied.

1. Experimental part

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An anodic coating of platinum with sodium cuprate was conducted in a 13 N solution of NaOH containing 10-13 g/l Cu(II) in the form of a cuprite solution. The cuprite solutions were prepared by means of dissolution of Cu(OH)₂ in alkali with successive centrifugation. A smooth platinum sheet with an area of 2 or 1 cm², which had been washed with nitric acid, served as the anode. The anodic region was separated from the cathodic region by a diaphragm. Electrolysis was conducted over the course of 20-30 minutes at room temperature at $D_x \approx 200 \mu\text{A}/\text{cm}^2$. As a result, the platinum anode was coated with dense orange cuprite, at a higher or lower current density and at a higher temperature, the quality of coating of trivalent copper is worse, or the coating is not obtained at all. For example, with an

¹In preceding works for the identification of anionic compounds, we used Werner nomenclature. In connection with this new project, the nomenclature of anionic compounds of trivalent copper in this work will be cuprate, for trivalent compounds, and cuprite, for divalent.

increase in current density, a more intense isolation of oxygen causes a deterioration of the coating. An insufficiently clean platinum anode is poorly coated with cuprate.

Upon coating of the platinum with barium cuprate, a mixture of 1 part saturated barium hydroxide solution at 40-50°C and 2 parts of the above described cuprite solution served as the electrolyte. Precipitates of barium cuprate are brownish-violet, more rusty, and binding with the platinum is weaker, than in the case of sodium cuprate. The composition of the electrochemically synthesized barium cuprate is studied in [11].

On copper, we succeeded in obtaining only barium cuprate. The coating was obtained upon anodic oxidation of a copper sheet (5 cm²) in a warm saturated solution of Ba(OH)₂ at $D_x \approx 0.04$ A/cm².

The obtained coating was washed several times with alkali solutions and placed in a cell for measuring the potential. The concentration and temperature of the alkali used for washing the coating was the same as in the measurement cell. Upon the use in the measurement cell of a less concentrated alkali solution (<1 N) a relatively porous coating of barium cuprate containing barium hydroxide in the pores and on the surface, and possibly, sodium hydroxide, was quickly washed in distilled water before washing in corresponding alkali solutions. In the opposite case, the pH of the layer near the electrode could not correspond with the pH of the solution.

For measurement of the dependence of potential on the concentration of Cu(II), freshly prepared solutions of a cuprite, Na₂CuO₂, were used.

A saturated calomel electrode served as the reference electrode, and the circuit was completed with a saturated KCl solution. Substitution of

potassium chloride in connection with a saturated solution of sodium nitrate has almost no effect on the results of the measurement. An addition of carbonates to the alkali does not affect the value of the potentials either, therefore the measurements were conducted in an open cell.

For evaluation of the value of diffusion potential, the potential of the circuit, composed of a mercury-mercuric oxide electrode was measured in a 1 N solution of NaOH, and a saturated calomel electrode with potassium chloride completed the circuit. It was found, that the potential of the mercuric oxide electrode was higher by 5-6 mV in its intrinsic potential (measured by a hydrogen electrode). Thus, the value of diffusion potential in this case is negligible.

Measurements of potential differences were conducted using the "Dupasar" pH/mV meter. The value of the scale division of the millivoltmeter is 2 mV, however the potential on the scale can only be fixed with a precision of up to 1 mV.

All experiments were repeated many times. It follows to note that the results of measurements depended to some degree on the quality of the coatings. If the platinum was only partially coated with the cuprate, the results obtained were decreased somewhat.

Experiments showed that upon mechanical contact of the platinum electrode with barium cuprate, the potential in 1 N NaOH is close to that measured in work [8] and is noticeably lower than the potential of the corresponding electrochemical coating.

A coating of sodium cuprate under conditions of lower alkalinity was dissolved well enough and the values of the potentials dropped. Upon

measurements of sodium cuprate potentials in NaOH solutions with a 75 concentration less than 4 N at a temperature of 5°C, and less than 4N at a temperature of 25°C, the potential dropped continuously. Upon work with a barium cuprate coating, a stable potential can be obtained in NaOH solutions with much lower concentrations.

The data presented in Fig. 1 shows that in alkaline NaOH solutions above 0.1 N, a stable potential is established more quickly in less alkaline solutions, and at a concentration of NaOH solutions of less than 0.1 N, the potential of barium cuprate is established more quickly in the more alkaline solution. Judging from the data presented below, this, apparently, is connected with the fact, that for an alkalinity of above and below 0.1 N, the potential determines different reactions. The stationary potential is established more quickly with the growth of temperature and upon addition of cuprite. Usually in the prestationary state, a growth in potential is observed, however, upon the addition of significant quantities of cuprite, the potential drops.

It was established that at the platinum and copper anodes, the values of stationary potentials for trivalent copper are the same: upon coating of the platinum with barium cuprate, the potential in a 1 N NaOH solution at 5°C is equal to 777 mV (by a hydrogen electrode), and upon coating of the copper electrode - 778 mV.

Upon addition of a small quantity of cuprite to the alkaline solution, the stationary potential hardly changes, upon a relatively significant addition, however, the dependence closely conforms with to the Nernst equation, and the changes correspond to a single electron transfer (Fig. 2,

curve 1).

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Such a dependence, most probably, was caused by the fact that in the potential determining electrode layer, aside from an addition of Na_2CuO_2 , there are anions of divalent copper, forming as a result of the reduction of trivalent copper. Their concentration could be computed based on the

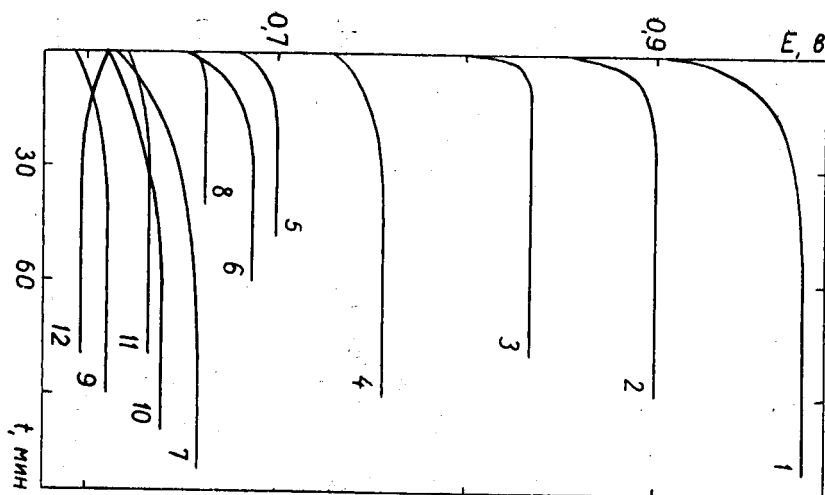


Fig. 1. Characteristic changes of the stationary potential over time. Barium cuprate, $t_0 - 5^\circ\text{C}$, alkalinity of the NaOH solution, N: 1 - 0.001, 2 - 0.01, 3 - 0.1, 4 - 2. Sodium cuprate, $t_0 - 5^\circ\text{C}$, alkalinity of the solution, N: 5 - 8, 6 - 10, 7 - 13; $t_0 - 25^\circ$, alkalinity of the solution, N: 8 - 10, 9 - 13. Sodium cuprate, $t_0 - 3^\circ\text{C}$, solution alkalinity 13 N, addition of $\text{Na}_2\text{CuO}_2 \cdot n\text{H}_2\text{O}$. 10 - 10^{-3} , 11 - 10^{-2} ,

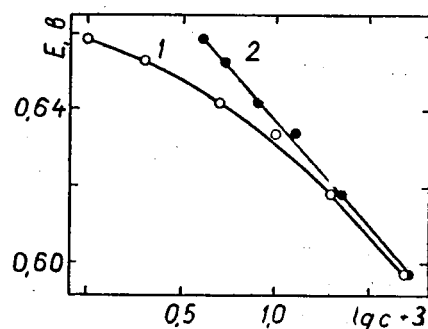


Fig. 2. Dependence of the value of the stationary potential of sodium cuprate in a 13 N NaOH solution at 5°C on: $+1c^* \text{Cu(II)}$
 $c_{\text{Cu(II)}}, 2 - c$

obtained experimental data, assuming, that for the value of this concentration and the ionic concentration of trivalent copper in the stationary phase of cuprite addition there is little effect.

On the basis of calculations, the following equation is set forth:

$$E = E_0 + \frac{2,303 RT}{n_1 F} \lg c_{\text{Cu(III)}}^* - \frac{2,303 RT}{n_2 F} \lg (c_{\text{Cu(III)}}^* + c_{\text{Cu(II)}}) - \frac{2,303 RT}{n_3 F} \lg a_{\text{OH}^-}, \quad (1)$$

where E is the measured stationary potential, $c_{\text{Cu(III)}}$ is the concentration in the stationary phase of trivalent copper anions, $c_{\text{Cu(III)}}^*$ is the concentration of divalent copper anions in the electrode layer, formed for trivalent, $c_{\text{Cu(II)}}$ is the concentration of the added divalent copper, a_{OH^-} is the hydroxyl ion concentration.

As occurs from the dependencies presented below, in an alkalinity range of 0.1-17 N NaOH, $n_2, n_3, z_2, z_3 = 1$.

This could mean that

$$E_0 + \frac{2,303 RT}{n_1 F} \lg c_{\text{Cu(III)}}^* = E_1. \quad (2)$$

Then

$$E = E_1 - \frac{2,303 RT}{F} \lg (c_{\text{Cu(II)}}^* + c_{\text{Cu(II)}}) - \frac{2,303 RT}{F} \lg a_{\text{OH}^-}. \quad (3)$$

From here, in the limits of a similar alkalinity for any two concentrations of added cuprite, for example $c'_{\text{Cu(II)}}$ and $c''_{\text{Cu(II)}}$, various stationary potentials will be observed

$$E' - E'' = \frac{2,303 RT}{F} \lg \frac{c_{\text{Cu(II)}}^* + c'_{\text{Cu(II)}}}{c_{\text{Cu(II)}}^* + c''_{\text{Cu(II)}}}. \quad (4)$$

Thus,

$$10^{\frac{(E' - E'') F}{2,303 RT}} = \frac{c_{\text{Cu(II)}}^* + c'_{\text{Cu(II)}}}{c_{\text{Cu(II)}}^* + c''_{\text{Cu(II)}}} \quad (5)$$

and reducing the value

$$c_{\text{Cu(II)}}^* = \frac{c'_{\text{Cu(II)}} - c''_{\text{Cu(II)}} \cdot 10^{\frac{(E' - E'') F}{2,303 RT}}}{10^{\frac{(E' - E'') F}{2,303 RT}} - 1}. \quad (6)$$

The values $c_{\text{Cu(II)}}^*$ were computed from equation (6) for various $c_{\text{Cu(II)}}$ pairs. By means of the substitution of a_{OH^-} into equation (3), the

average values of $c^*Cu(II)$, $cCu(II)$ and measured values of E corresponding to them, were obtained and are presented in this table of E_1 .

As is evident from Fig. 2, curve 2, the dependence of the measured potential on the total quantity of divalent copper in the electrode /// layer conforms to the Nerst equation. Thus, the calculations and above presented discussions are confirmed.

The value of E_1 is an indicator of the concentration of cuprate anions in the stationary phase. There obtained values are found in complete agreement with laws, which coincides with the stability of cuprates: the solubility and stability of sodium cuprate are increased with a decrease in alkalinity [12], barium cuprate, however, is practically insoluble, even in very weakly alkaline solutions, and in connection with this it is more stable.

Table 1. Results of computation of $c^*Cu(II)$ and E_1 by equations (6) and (3)

1 Покрытие	2 NaOH, н.	3 $c_{Cu(II)}$ в ммоль/л	4 E в в (при 5° C)	5 $c^*_{Cu(II)}$ в ммоль/л	6 E_1 в в
6 Купрат натрия	13	50	0,597	1,3	0,640
		20	0,618	0,5	0,639
		10	0,634	7,6	0,639
		5	0,642	3,1	0,640
		2	0,653	2,5	0,641
		1	0,659		0,640
		8 среднее		3	0,640
	10	50	0,643		0,664
		5	0,681	7,5	0,664
		2	0,688	6,5	0,664
		8 среднее		7	0,664
7 Купрат бария	6	10	0,669		0,612
		5	0,684	0,69	0,612
		1	0,713	0,72	0,612
		8 среднее		0,7	0,612

Key: 1 - Coating; 2 - NaOH, N; 3 - in moles/l; 4 - E in V (at 5°C); 5 - E₁ in V; 6 - Sodium nitrate; 7 - Barium nitrate; 8 - average

Substituting the average values of E₁ into equation (3), the values of the potential at $c_{Cu(II)} = 0$ can be computed. The calculated values of the potential of sodium cuprate in the absence of additions of cuprite in a 13 N solution of NaOH are equal to 0.779 V, in a 10 N solution - 0.783 V, barium cuprate in a 6 N NaOH solution is, respectively, 0.786 V (at 5°C). Thus, in all three cases very similar values were obtained. This data gives evidence about the fact that in the stationary phase the relationship of concentration $c_{Cu(II)}/c_{Cu(II)}$ is almost a constant value, not 78

Table 2. Values of stationary potentials of cuprates in solutions with different alkalinities

NaOH, / н.	a_{OH^-}	2 Е в е купрата	
		Na	Ba
3 При 5° С			
13	112,45	0,660	
10	40,90	0,685	
8	18,72	0,701	
6	8,34	0,716	0,722
4	3,66	0,739	0,740
2	1,39		0,757
1	0,67		0,777
10 ⁻¹	0,077		0,831
10 ⁻²	0,01		0,904
5 · 10 ⁻³	0,005		0,928
10 ⁻³	0,001		0,984
4 При 25° С			
17	246,50	0,545	
13	87,10	0,620	
10	34,50	0,649	
4	3,66		0,708
1	0,68		0,746

Key: 1 - NaOH, N; 2 - E in V for cuprate; 3 - At 5°C; 4 - At 25°C

depending on the alkalinity of the solution and the solubility of trivalent copper compounds. This constancy is confirmed by data of the dependence of the potential on the concentration of hydroxyl ions (Fig. 3), and also in particular in the fact, that the established values of the stationary potentials of sodium cuprate and relatively insoluble barium cuprate in solutions of the same alkalinity (without additions of Na_2CuO_2) are the same (Table 2). The last fact, incidentally, shows, that in both cases the potential determines similar ions. Therefore, it is likely that the chemical structure of sodium and barium cuprate are the same.

Since the reduction of trivalent copper is an irreversible and uninterrupted process, then attainment of a stable potential and the mentioned constancy $c_{\text{Cu(III)}}/c_{\text{Cu(II)}}$, noted in Fig. 1, is explained by the fact that the increase in the electrode potential-determining layer $c^*_{\text{Cu(II)}}$ is limited in the stationary phase by the diffusion of divalent copper into the bottom of the solution.

From Fig. 3 it is evident, that the measured potential is a linear function of the pH with a different slope coefficient of alkalinity in various intervals. Using data of Table 2 for calculations, we obtain the following dependencies.

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At 5°C in the alkalinity range of 0.1-13 N NaOH (section AB in Fig. 3) has the dependence:

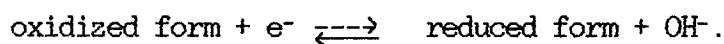
$$E = 0,77 - 0,052 \lg a_{\text{OH}^-}. \quad (7)$$

As we see, the data of equation (7) approximately corresponds to the above calculated values of the potential at $c_{\text{Cu(II)}} = 0$.

At 25° in the alkalinity range of 1-17 N NaOH the dependence in analogous to (7):

$$E = 0,74 - 0,060 \lg a_{\text{OH}^-} \quad (8)$$

Thus, in the indicated alkalinity range, a half reaction of the following type is observed:



In the alkalinity range 0.001-0.1 N NaOH (section BC in Fig. 3) the dependence of the potential on pH (at 5°C) is expressed by the equation

$$E = 0,74 - 0,081 \lg a_{\text{OH}^-}. \quad (9)$$

Here the slope coefficient already corresponds to $3/2 \cdot (2.303RT/F)$ and has the following half reaction type:

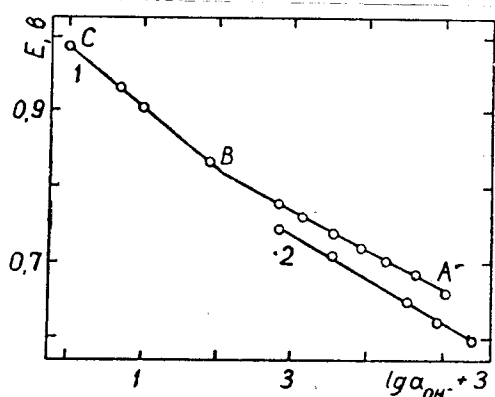
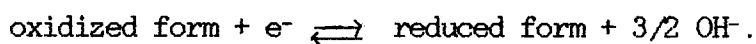


Fig. 3. Dependence of the stationary potential (overall for sodium and barium cuprates) on the alkalinity of the solution at: 1 - 5°C, 2 - 25°C

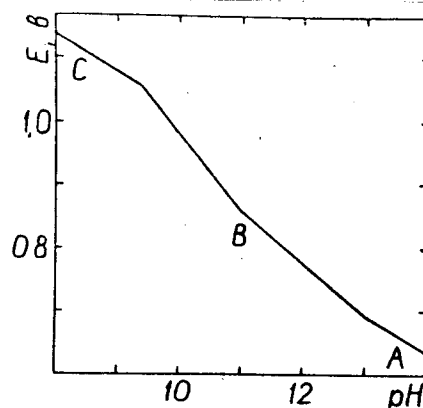


Fig. 4. Diagram E-pH from data of this work - AB, work [7] - BC

If the experimental equation (9) is recalculated for 25°C, then on the basis of the dependencies found by Columbo [7]:

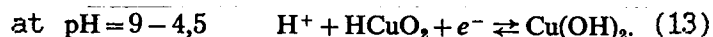
$$\text{at pH} = 11 - 9 \quad E = 2.28 - 0.120 \text{ pH}, \quad (10)$$

$$\text{at pH} = 9 - 4.5 \quad E = 1.72 - 0.060 \text{ pH} \quad (11)$$

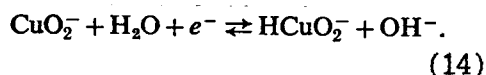
and our data can lead to the general diagram E-pH (Fig. 4), which shows that in principle the data of [7] and this work complement each other. In view of the fact that in the cited work the dependencies presented were for periodate and tellurate complexes of trivalent copper, Fig. 4 confirms the opinion [7, 13] that these complexes dissociate to oxo- compounds in solution (the dissociation is very weak), the ions of which are potential

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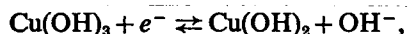
determining. By the equations (10) and (11) determined by Columbo, the potential determining reactions are described:



By half reactions (7) and (8) the half reaction, apparently, can be expressed in the form

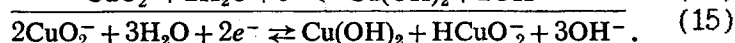
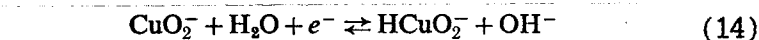


Naturally, the half reaction mechanism does not have a place [7, 8] in this case



since it is known that in such concentrated solutions of alkali, divalent copper dissolves relatively well and is found in solution in the form of an anionic compound, that is, cuprite. The data presented in this work on the effect of additions of Na_2CuO_2 on the oxidation-reduction potential confirms the participation in the electrode reaction of such types of anions. Inasmuch as with an increased valence of the metal the acidic properties of hydroxide are strengthened, the combination of trivalent copper should be more so with anionic compounds.

In 10^{-3} - 10^{-1} N solutions of NaOH, the potential can determine the equation, derived from equations (14) and (12):

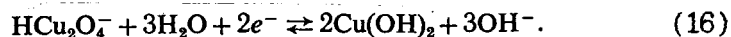


Thus, at first glance, there is a place for a regular series of reactions: at 4.5 - 9 pH the potential determines the system of acidic cuprate - copper hydroxide, at a pH of 9 - 11 - cuprate - copper hydroxide, at pH > 13 - cuprate - acidic cuprite, and in the interval between the last two ranges of alkalinity, a mixture of reactions, occurring in these two ranges.

However, the strict linearity of the section BC excludes a mixed reaction, as in this case on the right part of the section we should find reaction (14) and the slope coefficient should be less than $(3/2) \cdot (2.303RT/F)$, and on the left part - reaction (12) with a larger slope coefficient.

A calculation of the free energy of the cuprate anion, derived from equations (14) and (12) serves as the basis for an objection against the above presented system of equations. The reactions were calculated from equation (14) with the use of the value of potential from equation (8) and data of [4], the value of the free energy of the CuO_2^- ion is equal to 25.2 kcal, at the time when the calculation from reaction (12) and equation (10) give the result, equal to 32.7 kcal. Such divergences are possible only due to error in the potential values in equations (8) or (10), exceeding 0.3 V. Similar errors are excluded (see Fig. 4). /81

We can assume, that in the alkalinity range of 10^{-3} - 10^{-1} N NaOH the potential determines the reaction type



This reaction is not mixed and in this meaning does not contradict experimental data. The oxidizing agent in this reaction is acidic dicuprate. In principle, it does not preclude that compounds $\text{BaO} \cdot \text{Cu}_2\text{O}_3$ and $\text{Na}_2\text{O} \cdot \text{Cu}_2\text{O}_3$ have a structure of the BaCu_2O_4 and $\text{Na}_2\text{Cu}_2\text{O}_4$ type and not that of $\text{Ba}(\text{CuO}_2)_2$ and NaCuO_2 . Then instead of equation (14) the potential can determine the reaction



However, if this is conducted analogously with properties of borates (in a weakly alkaline medium - polyborates $\text{B}_4\text{O}_7^{--}$, in strongly alkaline - BO_2^-), then in a strongly alkaline medium the oxidizing agent is CuO_2^- , and equation (14) remains in force. Generally, there is no principle difference between these two reactions.

It is also possible, that equations (10) and (11) do not describe reactions (12) and (13), but some other reaction.

Thus, it is impossible to give a single answer on the potential determining reactions of the studied system.

Experiments on the determination of the effect of temperature on the potential of of cuprate, conducted in a temperature range of 5-25°C, showed that the temperature dependence is linear (Fig. 5).

From equations (7) and (8) it follows that

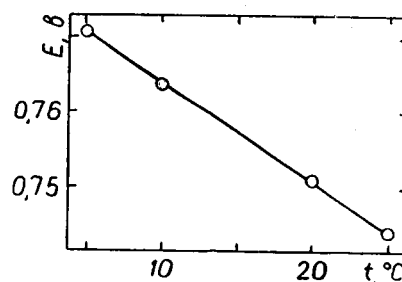


Fig. 5. The effect of temperature on the stationary potential of sodium cuprate. Data was obtained in a 10 N NaOH solution and was reread to $\text{aOH}^- = 1$.

(18)

$$E = 0,74 - 1,5 \cdot 10^{-3} (t^{\circ} - 25^{\circ}).$$

2. Conclusions

1. The dependence of the potential of sodium and barium cuprate coatings on platinum and copper on the alkalinity of solution and additions of cuprite was studied.

2. In 17 - 0.1 N solutions of NaOH, a linear dependence between the stationary potential of cuprate and the pOH with a slope coefficient of $(2.303) \cdot (RT/F)$, and at 10^{-1} - 10^{-3} N NaOH - with a slope coefficient of $(3/2) \cdot (2.303 \cdot RT/F)$ was established.

3. Equations were presented, showing that the effect of cuprite additions at the stationary potential conform to the Nernst equation.

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16. Abstract Potentials of barium and sodium cuprate coatings, obtained by an electrochemical method on platinum, and in isolated cases, on copper, were measured. It was shown that the stationary potentials were the same in the case of both cuprates. In 17-0.1 N NaOH solutions, the following dependencies were established: at 5°C $E = 0.77 - 0.52 \lg a_{OH^-}$, at 25°C $E = 0.74 - 0.060 \lg a_{OH^-}$. In 10^{-1} - 10^{-3} N NaOH solutions, the slope coefficient for a linear dependence of the potential on pH is equal to $(3/2) \cdot (2.303RT/F)$. Upon addition of cuprite, the measured stationary potential complies with the dependence $E = E_1 - (2.303RT/F) \lg a(c_{Cu(II)}^* + C_{Cu(II)}) -$ $(2.303RT/F) \lg a_{OH^-}$, where $c_{Cu(II)}$ is the concentration of the added divalent copper, and $c_{Cu(II)}^*$ is the concentration of the species formed upon reduction of trivalent copper. Possible variations of potential determining reactions were discussed.					
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